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TRIBOLOGICAL IMPROVEMENT AND CHARACTERIZATION OF HIP PROSTHESES WITH NANOSTRUCTURED SURFACES USING ADVANCED MICROTECHNOLOGIES AND ATOMIC FORCE MICROSCOPY

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The main objectives of this scientific work include studying and improving tribological properties of hip prostheses in order to increase the functionality and life of the hip joint implants, and especially to adapt to new achieving methods of hip prostheses.Hip prostheses may be used for different periods of time, depending on their composition and micromechanical properties. Due to difficult conditions imposed by human body and huge demands, its durability is generally limited to 15-16 years. They deteriorate due to the high superficial pressures produced by mechanical movements of the body. Beside this, hip implants heat up due to friction during continue activities, leading to thermal damage and loss of implants fixation. The methods and instruments for this development are used from different branches of science and technique through incorporating nanostructured coatings in the design. The study of tribological properties and characterization of hip prostheses is realized by advanced microtechnologies and atomic force microscopy.

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1. Introduction

Human body's motor apparatus is composed of elements which allow mechanical interactions between human body parts and environment.

The joints are included in the interstitial elements system and are designed for transmitting fundamental components of the whole motor system's movement. Joint is a system with passive components (such as articular ends of bones, cartilages, ligaments, synovial fluid) and active components (muscles). The joint is a moving structure, but the ligaments are intended to limit mechanically the potential movement, to steer the surfaces in contact and to stiffen the joint.

The hip is essentially a ball and socket joint, formed by the articulation of the rounded head of the femur and the cup-like acetabulum of the pelvis. It forms the primary connection between the bones of the lower limb and the axial skeleton of the trunk and pelvis. The large head of the femur attaches directly to the acetabulum. The head of the femur is attached to the shaft by a thin neck region that is often prone to fracture in the elderly, mainly due to the degenerative effects of osteoporosis.

Hip joint's biomechanics requires an inherent stability through its design and due to the spherical shape. In the joint, large forces are generated by the strong periarticular muscles, which balance the body's weight amplified by the bone levers. Any flaw in the joint mechanism changes the intra-articular forces distribution, causing degenerative changes.

Normal function of joints may be disturbed due to illness and under the negative influence of other factors determined by the mode of human life and professional activity. In such cases the drug treatment does not give positive results, so the only way to alleviate pain, to restore limb

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length and joints mobility is pseudo-joints implantation. Arthroplasty surgery is a modern method of treatment for advanced stages of joints disease. The operation consists in the regeneration of the joints fragments damaged by disease using pseudo-implants.

A total hip prosthesis (THP) consists of three parts (Fig. 1):

- a cup that replaces the hip socket;
- a ball that replaces the fractured head of the femur;
- a stem that is attached to the shaft of the bone to add stability to the prostheses.

Joint component of femoral prosthesis is a piece of metal sphere made from Ti alloys, CoCr alloys or ceramic (aluminum oxide or zirconium oxide). Titanium and its compounds are commonly used due to their low density, high mechanical strength, good corrosion resistance and biocompatibility. For these reasons it is an alloy widely used for advanced biomedical applications. The material used to manufacture cups is high molecular weight polyethylene (UHMWPE), but there are also metallic or ceramic cups. Prosthetic head and polyethylene cup with much larger diameter and thicker wall lead to the formation of a friction torque increased by the radius difference of components. Cup having large outdoor diameter decreases the pressure per unit area, reducing the chances of bone resorption and loosening chances.

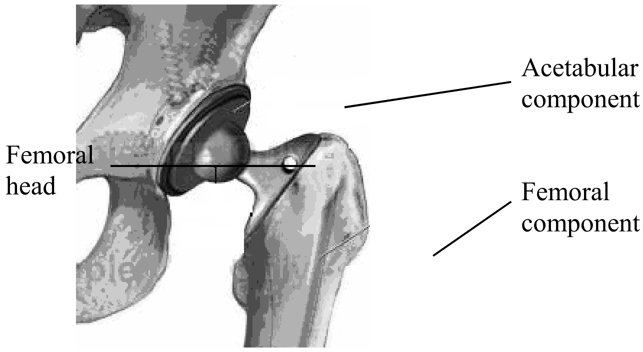


Fig. 1. Components of a total hip prosthesis.

Total hip prostheses function without problems in the human body for over 12-15 years. Their durability is limited by the wear resistance of the joint bearing femoral head - acetabular cup to mechanical, thermal, chemical and biological factors acting on it.

2. Wear mechanisms

The most common failures of hip prostheses are surgical problems (e.g., problematic orientation or problems in wound healing); host abnormalities or diseases (e.g., osteopenia); infections; material fractures, wear, and corrosion. The hip prostheses surfaces mainly deteriorate due to the high superficial pressures produced by mechanical movements of the body. The deterioration process has a complex mechanism, combining abrasive wear, adhesive wear, third body wear and fatigue wear [1]. This wear may cause loosening of the prostheses by the resulting poor mechanical force between the ball and socket of the hip.

After exclusion of hip prostheses from the human body, it was observed numerous scratches. Such scratching may be attributed to wear resulting in loss of material or plastic deformation of the surface without loss of material. Scratching of the metallic femoral head by wear particles (bone, cement, or metallic debris) [1] influence the hip prostheses stability. Particles smaller than 1 μm are the most active ones from a biological point of view [2], [3]. The scratches occurring on femoral head are considered as main cause of increased intensity of wear rate [4].

Expanding surgical procedure for total hip replacement and in younger patients, with intense physical activity, is required to find technical and technology solutions to increase the lifespan of total hip prosthesis.

3. Improvement methods

So far it has not succeeded completely removing the problems associated with the use of hip prostheses, i.e. loosening and fracture, rejection physiological reactions of the body and the most important, material wear. Therefore, it is necessary to realize a resistant prosthesis, with anticorrosive composition and high mechanical properties.

Stainless steel, titanium alloy, polymers, and ceramic composites undergo degradation after 10–15 years of use. For this reason you need to take into account the physiological loads placed on the implants. Material choices also must take into account immune system biocompatibility, the environment, corrosion issues, friction and wear of the articulating surfaces, implant fixation either through osseointegration or bone cement.

Metallic materials are relatively soft, but resistant to breakage. Metallic biomaterials have different influences on the human body, is distinguishing different forms of biological reactions, according to: concentration of metal, exposure time and route of administration. Zirconium alloy substrate is relatively soft when compared with cobalt-chrome alloy femoral heads and may deform in contact with acetabular shell materials in the case of dislocation. Ceramics, with extremely low wear rates, are undisputed, but there remains a risk of catastrophic failure because of the inherently low cracking toughness of the materials. Oxinium components have a unique ceramic layer (4 μm thick) to mitigate the wear, it is related to a zirconia ceramic material, and is used on an integral metal substrate that provides toughness and high fatigue strength.

Generally, pure metals are rarely toxic. Toxic and allergenic effects depend on the concentration and nature mixtures (oxides, simple or complex salts), and two compounds of the same metal can induce strong responses, but different.

Taking into account the above, the international scientific community has made and still makes efforts to increase hip prostheses durability. In order to improve mechanical properties of hip prostheses, these have been coated with different materials, which have superior properties.

Metal–matrix composite (MMC) coatings reinforced with hard ceramic particles are promising materials for improvement of mechanical properties over conventional monolithic alloys.

A thin hydroxyapatite (HA) coating, having the composition similar to bone apatite has an important property to achieve rapid biological connections in bone structures. Applied on textured metal surfaces, combines the mechanical attributes of metals with osteoconductive properties of metals and with the biocompatibility of ceramics. HA coatings on metallic substrates suffer from many problems like cracking and peeling off [5], which result in the release of harmful metal ions to the body's environment. The adherence of the HA coating to the substrate is very poor. In order to overcome inferior adhesion an interlayer coating can be provided in between the metal substrate and the HA outer layer [8].

Raw nanomaterials – which include nanoparticles and nanocrystalline materials that are readily manufactured and can substitute less performant bulk materials - can be used as biocompatible materials or coatings in prostheses, and implants.

There are several methods of thin and multilayer coating of the femoral heads, suitable of the coating material and the substrate. The most common PVD coating process is suitable both Co-Cr-Mo alloy, and Ti-6Al-4V. Since titanium nitrides are hard biocompatible materials with excellent resistance to abrasion, have been developed more advanced processing methods in order to achieve a nitrided layer on the surface of materials.

However, the compounds have poor tribological characteristics during dry foil, including a high friction coefficient, low wear resistance and high susceptibility to seizures. The tribological properties of these alloys are known to be weak, especially in abrasive and sliding conditions. High friction coefficient and severe adhesive wear occurs frequently when Ti-6Al-4V alloy is sliding against other engineering materials.

So, depending on their composition (ceramic, metals) hip prostheses may be used for different periods of time.

4. Experimental studies

Wear processes changes are often impossible to see with the naked eye.

A methodology of ascending degrees of resolution was established using macroscopic (resolution millimeters), microscopic (resolution microns) and nanoscale (resolution nanometers) measurements. For these reasons, we are trying to improve the tribological properties of hip prostheses by structural changes using nanostructured coatings and to characterize the surface of a microscopic technique.

This scientific paper presents an analysis regarding the topography and tribological parameters of femoral heads structures and of femoral heads coated with TiN. It was analyzed a series of total hip prosthesis with modular femoral head of Ti-6Al-4V alloy and Co-Cr alloy PVD coated with TiN, recovered following revision surgery, and were identified scratching, cracking, peeling and tribocorrosion.

Femoral heads were first photographed, investigated in terms of deviations and its surface condition was investigated by optical microscopy, using an atomic force microscopy NanoLaboratory AFM Probe NTEGRA NT - MDT, Russia (Figure 2), of institute's own equipment, working in the noncontact mode. AFM images were processed using Nova SPM software. In this way was obtained the roughness of the studied surface and were calculated other tribological parameters.

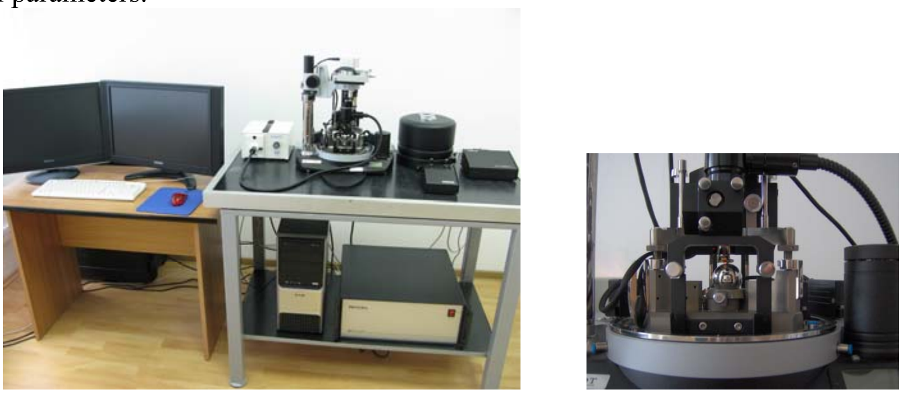


Fig. 2. Atomic Force Microscope NTEGRA Probe NanoLaboratory (a); detail of an AFM measurement (b)

5. Results

The optical microscopy study of the femoral head, made before coating, revealed that 25% of the entire surface has isolated micro-scratches despite of polished macroscopic aspect. It was identified an embedded wear particle which scratched the femoral head surface (Fig. 3a). The large scratch could be related to the abrasion due to third body, namely the wear particles. Signs of severe micro cutting were detected also in another region (Fig. 3b). In Fig. 3c one can observe small scratches and pits produced by biotribological corrosion. The corrosion pits occurred due to strong mechanical loading combined with the local temperature increase, at the contact interface.

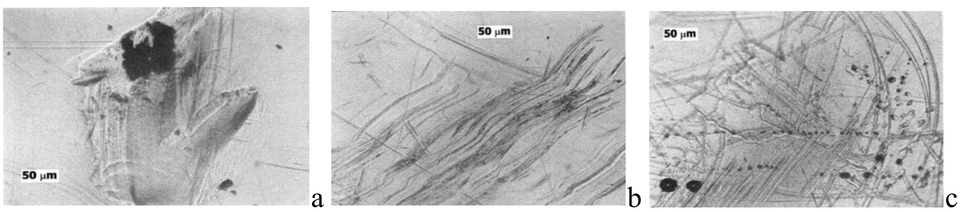
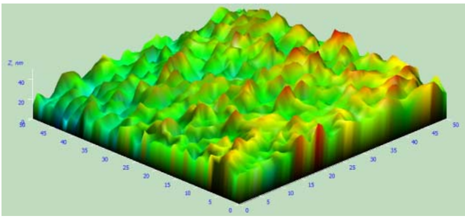


Fig. 3. Scratching (a), micro cutting (b) and corrosion fretting (c) of femoral head

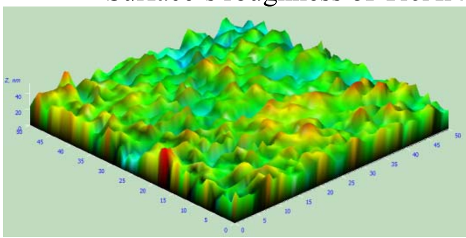
Similar results were obtained also for the others parts of the prosthesis. Three special areas were observed on the femoral head made from Ti. There are regions with a polished visual aspect and areas with a small level of luminosity. In the second part appears a high deterioration degree, with a lot of scratches. The third category of regions covers more than 50% of the femoral head and has an intermediate level of luminosity. Here, the deterioration level is not so high.

Using AFM measurements it can be determined exactly the surface's roughness and can be observed all the surface bumps (Fig. 4, Fig. 5). As it is shown in the following examples, the roughness has different values (in different parts of the same femoral head), depending on the movements of the body that uses the prostheses.



Amount of sampling	2601
Max	51.3313 nm
Min	0 nm
Peak-to-peak, Sy	51.3313 nm
Ten point height, Sz	27.5242 nm
Average	29.5981 nm
Average Roughness, Sa	5.65844 nm
Second moment	30.4287
Root Mean Square, Sq	7.06102 nm
Surface skewness, Ssk	-0.0889571
Coefficient of kurtosis, Ska	-0.0545433
Entropy	5.029
Redundance	-68.6369

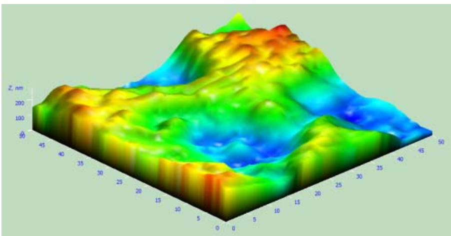
Surface's roughness of Ti6Al4V femoral head



Amount of sampling	2601
Max	53.1167 nm
Min	0 nm
Peak-to-peak, Sy	53.1167 nm
Ten point height, Sz	26.0182 nm
Average	28.2611 nm
Average Roughness, Sa	6.24355 nm
Second moment	29.3228
Root Mean Square, Sq	7.81915 nm
Surface skewness, Ssk	-0.280135
Coefficient of kurtosis, Ska	-0.0182407
Entropy	5.13756
Redundance	-67.8078

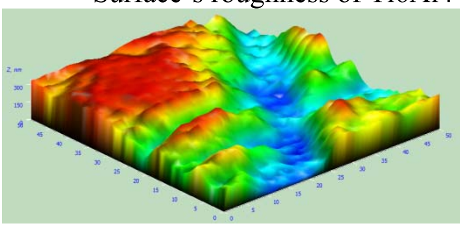
Surface's roughness of CoCr femoral head

Fig. 4. Femoral head surface's roughness measured using NTEGRA Atomic Force Microscopy



Amount of sampling	2601
Max	286.397 nm
Min	0 nm
Peak-to-peak, Sy	286.397 nm
Ten point height, Sz	140.899 nm
Average	142.588 nm
Average Roughness, Sa	58.8148 nm
Second moment	157.995
Root Mean Square, Sq	68.0512 nm
Surface skewness, Ssk	-0.280579
Coefficient of kurtosis, Ska	-1.12596
Entropy	6.80471
Redundance	-17.7285

Surface's roughness of Ti6Al4V femoral head



Amount of sampling	2601
Max	352.544 nm
Min	0 nm
Peak-to-peak, Sy	352.544 nm
Ten point height, Sz	176.736 nm
Average	207.335 nm
Average Roughness, Sa	88.7965 nm
Second moment	230.829
Root Mean Square, Sq	101.461 nm
Surface skewness, Ssk	-0.190727
Coefficient of kurtosis, Ska	-1.2669
Entropy	7.0297
Redundance	-15.1352

Surface's roughness of CoCr femoral head

Fig. 5. Femoral head surface's roughness measured using NTEGRA Atomic Force Microscopy

At the nitriding in plasma, nitrogen atoms diffuse into the titanium matrix, forming a layer of TiN and Ti_2N compounds, usually followed by a deeper diffusion layer.

Scratching tests were performed in these areas, using different values of the acting forces. Finally, these surfaces show signs of damage visible to the naked eye. After characterization of these surfaces using AFM has been observed that the damage and roughness values depend on the amount of force applied. In this phase of the investigation we found that there are signs of peeling of the TiN coating. It highlights the loss of coating adhesion, crushing them, embedding the resulting particles in acetabular cup and the failure of total hip replacements. It is possible that one of the causes of loss of adhesion is the lack of coverage to match the mechanical characteristics of coating and substrate, probably in the main modules of elasticity. It shows that the lack of a suitable adhesion layer – sub-layer is the main cause of failure of coatings femoral heads.

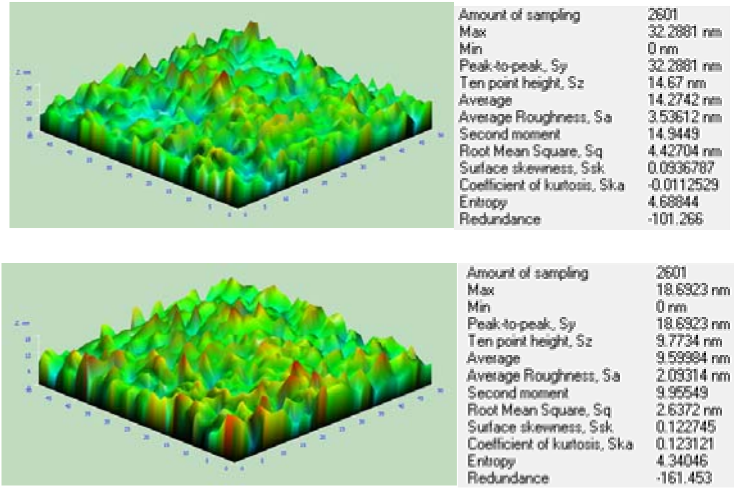


Fig. 6. Roughness of femoral head surface with TiN coating measured using NTEGRA Atomic Force Microscopy

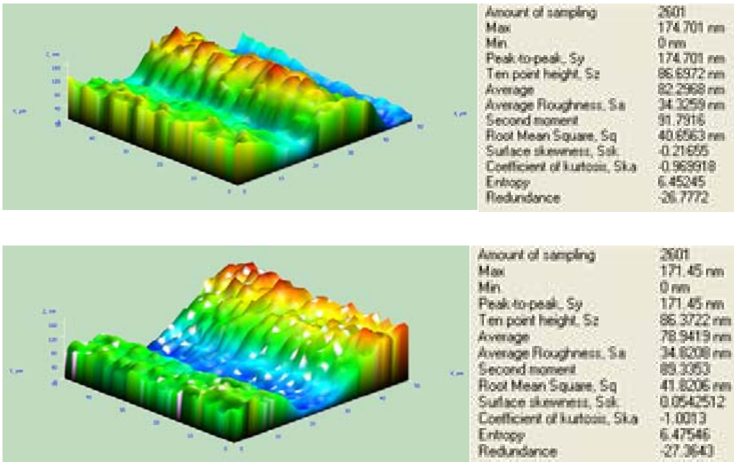


Fig. 7. Roughness of femoral head surface with TiN coating after scratching test using NTEGRA Atomic Force Microscopy

Another important result was that the TiN-coated surfaces show a roughness value lower than uncoated surfaces. It was also noted that the formation of a titanium oxide layer on the surface, lead to a lower value of friction.

Taking into account all these studies we shall continue the research and AFM microscopy studies. These could improve topographic characterization of nanostructured coatings surface.

6. Conclusion

As it was demonstrated in the last years, femoral head damage may occur during hip joint movements and may lead to accelerated materials wear. This is an important reason for femoral head coating, which offer the opportunity to improve system's properties. In order to obtain a clear characterization of the coatings the study of its topography is useful.

PVD TiN coatings offer a greater surface hardness to the femoral heads, but the lack of adherence or poor adherence on long term, in the strong mechanical loading conditions, make it deformed, crack, crumble and become a factor for increased abrasion.

We believe that the essential factor for increasing the durability of femoral heads with thin coatings is ensuring of a high adherent coating on the substrate. Machining, substrate treatment and its roughness, together with tribological parameters of the PVD coating has to be chosen in order to facilitate the establishment of the double diffusion interlayer between layer and substrate.

Taking into account that nanostructured coatings offer the opportunity to improve system's properties, the next steps will be based on these experiments and some other materials will be used and characterized to protect against hip prosthesis breakdown.

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